



Attorney Docket No.: 056203.50314US  
PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: SHUICHI KANNO ET AL  
Serial No.: 09/005,006      Group Art Unit: 1754  
Filed: JANUARY 9, 1998      Examiner: Ngoc Yen M Nguyen  
Title: PROCESS FOR TREATING FLUORINE COMPOUND-  
CONTAINING GAS

REPLY TO OFFICE ACTION

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TC 1700

Mail Stop: RCE  
Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

Sir:

This is in reply to the Final Office Action mailed April 9, 2003. This reply  
is accompanied by a Request for Continued Examination under 37 CFR 1.114.

Kindly enter the following amendment.

IN THE CLAIMS

1 – 39. (Cancelled)

40. (Currently Amended) A process for treating a fluorine compound-containing gas, comprising:

contacting a gas stream containing at least one compound consisting of (a) carbon and fluorine, (b) carbon, hydrogen and fluorine, (c) carbon, hydrogen, oxygen and fluorine, (d) SF<sub>6</sub>, and (e) NF<sub>3</sub>, wherein the concentration of the fluorine compound is 0.5 to 10% by volume, with a catalyst comprising alumina as an active compound and 7.2 to 49.4 wt.% of nickel oxide, said catalyst containing a composite oxide of aluminum and nickel;

adding steam or a reaction gas containing steam and oxygen to the gas stream; and

effecting a hydrolysis reaction between the at least one compound and the steam, thereby producing a treated gas containing hydrogen fluoride.

41. (Previously Presented) A process according to Claim 40, further comprising washing the treated gas with water to remove the hydrogen fluoride.

42. (Previously Presented) A process according to Claim 40, further comprising washing the treated gas with an alkaline solution or slurry to neutralize the hydrogen fluoride and other acidic compounds.

43. (Previously Presented) A process according to Claim 40, further comprising washing the treated gas with water and subsequently neutralizing

the water that has absorbed the hydrogen fluoride with an alkaline solution or slurry.

44. (Currently Amended) A process according to Claim 40, wherein the catalyst further comprises ~~7.2 to 61.3% by weight of zinc oxide, and wherein a~~ ratio of aluminum to a total of nickel and zinc is 50 to 99: 50 to 1 by atom.

45. (Previously Presented) A process according to Claim 40, wherein the catalyst consists essentially of alumina and nickel oxide and a composite oxide of aluminum and nickel.

46. (Previously Presented) A process according to Claim 40, wherein the at least one compound is at least one compound selected from the group consisting of CF<sub>4</sub>, C<sub>2</sub>F<sub>6</sub>, C<sub>3</sub>F<sub>8</sub>, C<sub>4</sub>F<sub>8</sub>, C<sub>5</sub>F<sub>8</sub>, CHF<sub>3</sub>, CH<sub>2</sub>F<sub>2</sub>, CH<sub>3</sub>F, C<sub>2</sub>HF<sub>5</sub>, C<sub>2</sub>H<sub>2</sub>F<sub>4</sub>, C<sub>2</sub>H<sub>3</sub>F<sub>3</sub>, C<sub>2</sub>H<sub>4</sub>F<sub>2</sub>, C<sub>2</sub>H<sub>5</sub>F, CH<sub>2</sub>OCF<sub>2</sub>, SF<sub>6</sub>, and NF<sub>3</sub>.

47. (Previously Presented) A process according to Claim 40, wherein the at least one compound is at least one compound selected from the group consisting of CF<sub>4</sub>, C<sub>2</sub>F<sub>6</sub>, C<sub>3</sub>F<sub>8</sub>, C<sub>4</sub>F<sub>8</sub>, C<sub>5</sub>F<sub>8</sub>, CHF<sub>3</sub>, CH<sub>2</sub>F<sub>2</sub>, CH<sub>3</sub>F, C<sub>2</sub>HF<sub>5</sub>, C<sub>2</sub>H<sub>2</sub>F<sub>4</sub>, C<sub>2</sub>H<sub>3</sub>F<sub>3</sub>, C<sub>2</sub>H<sub>4</sub>F<sub>2</sub>, C<sub>2</sub>H<sub>5</sub>F, SF<sub>6</sub>, and NF<sub>3</sub>.

48. (Currently Amended) A method of treating a gas containing a perfluoro-compound, comprising:

contacting the gas containing a fluorine compound in a concentration of 0.5 to 10% by volume at a temperature of 400 to 800°C with a catalyst comprising aluminum oxide as an active component and 7.2 to 49.4 wt.% of nickel oxide, said catalyst containing a composite oxide of aluminum and nickel, in the presence of steam, whereby the perfluoro-compound is decomposed by hydrolysis to produce a treated gas containing hydrogen fluoride and acidic compounds; and

contacting the treated gas with water to absorb the hydrogen fluoride and the acidic compounds from the treated gas.

49. (Previously Presented) A process according to Claim 48, wherein the perfluoro compound is at least one compound selected from the group consisting of  $\text{CF}_4$ ,  $\text{CHF}_3$ ,  $\text{C}_2\text{F}_6$ ,  $\text{C}_3\text{F}_8$ ,  $\text{C}_4\text{F}_8$ ,  $\text{SF}_6$  and  $\text{NF}_3$ .

50. (Previously Presented) A process according to Claim 48, wherein the catalyst further comprises zinc oxide, the balance being aluminum oxide.

51. (Previously Presented) A process according to Claim 48, wherein the catalyst consists essentially of alumina and nickel oxide and composite oxide of aluminum and nickel.

52 – 74. (Cancelled)

75. (Withdrawn) A process according to claim 40, wherein the compound in the gas stream is  $\text{SF}_6$ .

76. (Withdrawn) A process according to claim 40, wherein the compound in the gas stream is carbon, fluorine and hydrogen.

77. (Withdrawn) A process according to claim 40, wherein the compound in the gas stream is  $\text{NF}_3$ .

78. (Withdrawn) A process according to claim 40, wherein the compound in the gas stream is at least one member selected from the group

consisting of  $\text{CHF}_3$ ,  $\text{CH}_2\text{F}_2$ ,  $\text{CH}_3\text{F}$ ,  $\text{C}_2\text{HF}_5$ ,  $\text{C}_2\text{H}_2\text{F}_4$ ,  $\text{C}_2\text{H}_3\text{F}_3$ ,  $\text{C}_2\text{H}_4\text{F}_2$ ,  $\text{C}_2\text{H}_5\text{F}$ ,  $\text{CH}_2\text{OCF}_2$ ,  $\text{SF}_6$  and  $\text{NF}_3$ .

79. (Previously Presented) A process according to claim 40, wherein the fluorine compound-containing gas to be treated is used as etchants or cleaners for semiconductors.

REMARKS

Claims 40, 44, and 48 are amended to further clarify the nature of Applicants' claimed invention. Support for the amendment to claim 44 can be found, for example, at page 10, lines 1 – 3 of the specification. Support for the amendments to claim 40 and 48 can be found, for example, at page 12, lines 19 – 22 and page 29, lines 15 – 17 (Example 6) of the specification.

The rejection of claim 44 under 35 USC 112 is rendered moot by Applicants' amendment, as the subject matter noted by the Examiner has been removed from the claim. Reconsideration and withdrawal of the rejection are respectfully requested.

The rejection of claims 40 – 43, 45 – 49, 51, and 79 under 35 USC 103(a) over Rossin et al., US Patent 6,069,291, optionally in view of Okazake et al., US Patent 5,151,263 and Imamura, US Patent 5,649,985, is respectfully traversed. Rossin et al. discloses catalytic treatment of low concentrations of perfluoroalkanes such as CF<sub>4</sub>, C<sub>2</sub>F<sub>6</sub>, etc., using aluminum oxide catalysts. Several elements, including nickel, may be added to the catalyst to stabilize it. However, Rossin et al. does not disclose or suggest catalysts that are effective for treatment of fluorine compounds in concentrations of 5000 ppm (0.5%) or higher, as required by the claimed invention. In particular, Rossin et al. provides no working examples at all of an aluminum oxide that also contains nickel.

Although no examples of a nickel-aluminum oxide catalyst are provided in Rossin et al., the Examiner notes the performance data from Example XVII in support of the rejection. Example XVII describes performance data for a zirconium-aluminum oxide catalyst, as described in Example XIV. This example

appears to have been selected based on the fact that it is the longest working example provided by Rossin et al. No other indication is given in the Office Action as to why the performance of a zirconium-aluminum oxide catalyst, or any of the other catalysts described in Rossin et al., is relevant in determining the patentability of a method involving the use of a nickel-aluminum oxide catalyst. Thus, the data in Example XVII does not appear to be relevant to the patentability of the methods of the claimed invention, which makes use of nickel-aluminum oxide catalysts.

However, even if performance data for the various catalysts disclosed in Rossin et al. is deemed to be relevant, such performance data does not support a rejection of the claimed invention. Example XVII discloses that a zirconium-aluminum oxide catalyst can convert 98% of a 400 ppm  $C_2F_6$  gas stream for up to 400 hours. By contrast, the method of the claimed invention allows for conversion of more than 99% of a gas stream containing 5000 ppm or greater of a fluorine containing gas for more than 2000 hours. (See Kanno declaration submitted February 7, 2002.)

To further demonstrate the unexpected nature of this result, Applicants have provided a second declaration from Mr. Kanno. In this declaration, Mr. Kanno details experiments to create the zirconium-cobalt-aluminum oxide catalyst of Example XVI. In spite of some difficulties in preparing the zirconium-cobalt-aluminum oxide catalyst, the catalyst was successfully synthesized. This catalyst was then used under the conditions recited in Example XVII. As shown in Figure 1, the zirconium-cobalt-aluminum oxide catalyst of Example XVI performed at least as well or better than the zirconium-aluminum oxide catalyst.

of Example XIV noted in the Office Action. The zirconium-cobalt-aluminum oxide catalyst was effective for conversion of over 99% of a gas stream containing 500 ppm of fluorine containing gas for time periods well in excess of 400 hours. (See Fig. 1, second Kanno declaration.) This represents a higher conversion rate over a longer period of time than the reported performance in Rossin et al. for the zirconium-aluminum oxide catalyst of Example XIV. However, at higher fluorine containing gas concentrations, the zirconium-cobalt-aluminum catalyst quickly lost its effectiveness. When used to treat a gas stream containing 5000 ppm of a fluorine containing gas, the conversion rate for the zirconium-cobalt-aluminum catalyst drops to below 40% in less than 200 hours. (See Fig. 2, second Kanno declaration.)

As demonstrated by the experimental results in the two Kanno declarations, the method of the claimed invention provides unexpectedly superior results relative to the methods described in Rossin et al. Rossin et al. provides no teaching or disclosure that the described catalysts are effective for treating gas streams containing 5000 ppm or more of a fluorine-containing gas. The experiments in the declarations also show that the benefits of the claimed invention are not merely inherent to the use of any of the broad range of catalysts disclosed in Rossin et al.

Additionally, the disclosures of Imamura and Okazake et al., either alone or in combination with Rossin et al., do not remedy the above shortcomings. For at least these reasons, reconsideration and withdrawal of the rejection to claims 40 – 43, 45 – 49, 51, and 79 is respectfully requested.



The rejection of claims 44 and 50 under 35 USC 103(a) over Rossin et al. in view of Rosenbaum, US Patent 5,460,792, is also respectfully traversed. In addition to failing to describe or suggest all of the limitations of the claimed invention, it is not even clear how or why the disclosures of Rossin et al. and Rosenbaum would be combined by one of skill in the art.

Claims 44 and 50 require decomposition of a fluorine-containing compound using a catalyst that contains aluminum oxide, nickel oxide, and zinc oxide. As described above, Rossin et al. fails to disclose or suggest a method for treating a fluorine-containing gas having a concentration of greater than 5000 ppm. Additionally, as noted by the Examiner, Rossin et al. does not disclose the use of zinc oxide. Rossin et al. also does not contemplate the use of carbonaceous catalysts. Rosenbaum describes carbonaceous catalysts for use in decomposing halogenated organic compounds. The carbonaceous catalysts are doped with various metals to provide catalytic activity. The organic compounds are then exposed to the doped carbonaceous catalyst at temperatures below 400°C. While Rosenbaum discloses use of zinc and nickel as a catalytic metal (Col. 13, line 59 to Col. 14, line 19), aluminum is not listed as a possible catalytic dopant. By contrast, Rossin et al. states that "aluminum oxide is the primary agent" for effecting the transformation of a perfluoroalkane. (Abstract) Additionally, Rosenbaum discloses use of the carbonaceous catalysts at temperatures of less than 400°C, while Rossin et al discloses temperatures between 400°C and 1000°C.

The Examiner asserts that the disclosure of both zinc and nickel in Rosenbaum provides the motivation to combine Rossin et al. with Rosenbaum.

Applicants respectfully disagree, as Rosenbaum does not even acknowledge the catalytic activity of aluminum, which is the primary material used in Rossin et al. Thus, one of skill in the art would not expect that the teachings of Rosenbaum could be applied to the invention of Rossin et al, especially given the different processing temperatures between the references.

Even if one of skill in the art would be motivated to combine Rosenbaum with Rossin et al., the combination would still not produce Applicants' claimed invention. Neither Rossin et al. nor Rosenbaum, either alone or in combination, provides a disclosure or suggestion of a method for treating a gas stream containing 5000 ppm or more of a fluorine containing gas. Thus, the combination of Rossin et al. and Rosenbaum would still fail to describe or suggest all of the features of the claimed invention. As a result, reconsideration and withdrawal of this rejection are respectfully requested.


In view of the foregoing amendments and remarks, the application is respectfully submitted to be in condition for allowance, and prompt, favorable action thereon is earnestly solicited.

If there are any questions regarding this amendment or the application in general, a telephone call to the undersigned would be appreciated since this should expedite the prosecution of the application for all concerned.

If necessary to effect a timely response, this paper should be considered as a petition for an Extension of Time sufficient to effect a timely response, and please charge any deficiency in fees or credit any overpayments to Deposit Account No. 05-1323 (Docket #056203.50311US).

Respectfully submitted,

November 10, 2003

  
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Attorney Docket No.: 056203.50311US  
PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: SHUICHI KANNO ET AL  
Serial No.: 09/005,006 Group Art Unit: 1754  
Filed: JANUARY 9, 1998 Examiner: Ngoc Yen M Nguyen  
Title: PROCESS FOR TREATING FLUORINE COMPOUND  
CONTAINING GAS

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TC 1700

REQUEST FOR CONTINUED EXAMINATION (RCE) TRANSMITTAL

Mail Stop: RCE

Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450

Sir:

This is a Request for Continued Examination (RCE) under 37 CFR §  
1.114 of the above-identified application.

1. Submission required under 37 CFR § 1.114

- a. ☐ Previously submitted
- i. ☐ Consider the amendment(s)/reply under 37 CFR 1.116  
previously filed on \_
- ii. ☐ Consider the arguments in the Appeal Brief or Reply  
Brief previously filed on \_
- iii. ☐ Other \_
- b. Enclosed
- i. ☒ Amendment/Reply
- ii. ☐ Affidavit(s)/Declaration(s)

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- iii. ☐ Information Disclosure Statement (IDS)
- iv. ☐ Other

2. Miscellaneous

- a. ☐ Suspension of action on the above-identified application is requested under 37 CFR 1.103(c) for a period of \_ months (not to exceed three months). The required fee under 37 CFR 1.17(i) is included in Section 3. a. iii. below.

- b. ☐ Other

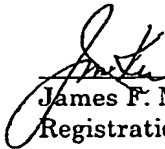
3. Fees

- a. ☒ The following fees are included in the payment indicated below:
  - i. ☒ RCE fee required under 37 CFR 1.17(e) (\$770.00)
  - ii. ☐ Extension of time fee required under 37 CFR 1.136 & 1.17
  - iii. Other \_
- b. ☒ Payment:
  - i. ☒ A check in the amount of \$770.00 is enclosed
  - ii. The Director is hereby authorized to charge the fees listed above in Section 3 a i-iii to the Deposit Account of Crowell & Moring, L.L.P., Account No. 05-1323 (Docket No. 056203.50311US)
  - iii. ☐ If a check should become detached or if there is any deficiency in fees, any necessary fees should be charged, or any overpayment in fees should be credited, to the Deposit Account of Crowell & Moring, L.L.P., Account No. 05-1323 (Docket No. \*).

If there are any questions regarding this amendment or the application in general, a telephone call to the undersigned would be appreciated since this should expedite the prosecution of the application for all concerned.

Respectfully submitted,

November 10, 2003

  
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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of

Shuichi KANNO et al.

Serial No. 09/005,006

Group Art Unit: 1754

Filed: January 9, 1998

Examiner: N. Nguyen

For: PROCESS FOR TREATING FLUORINE COMPOUND-  
CONTAINING GAS

D E C L A R A T I O N

COMMISSIONER FOR PATENTS  
Washington, D. C. 20231

Sir:

I, Shuichi Kanno, a Japanese citizen residing  
at 17-13, Ishinazaka-cho, 1-chome, Hitachi-shi, Ibaraki,  
Japan do hereby solemnly and sincerely declare THAT:

I graduated from the graduate course of  
Engineering Department (majoring in material chemistry)  
of Tohoku University in March, 1992;

I began employment with Hitachi, Ltd., the  
assignee of the above-identified application in April,  
1992 and have been engaged in said company, since that  
time, in the study of catalysts for chemical reactions;

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I am the first-named inventor of the above-identified application and am well aware of the prosecution history thereof;

I conducted the following Experiments in order to know how long the catalyst of Rossin et al (US Patent No. 6,069,291) can maintain high conversion rate, that is, the durability of the catalyst of Rossin et al.

[PREPARATION OF CATALYST]

A catalyst comprising aluminum oxide, cobalt and zirconia was prepared by the method disclosed in Example XVI of USP 6,069,291.

In a 5-liter beaker, 1.5 liters of deionized water was placed and stirred with a laboratory scale mixer. Then, 1001.16 of pseudoboehmite (G250, manufactured by Alcoa Inc.) was added to the stirring deionized water. Since the stirring became impossible during the addition of deionized water, 500 ml of deionized water was further added to conduct stirring again.

(Reasons for stopping of stirring seemed to be an increase of viscosity caused by polymerization. The addition of deionized water was based on my original idea derived from abundant experiments.)



After addition of whole amount of the pseudo-boehmite, the stirring became almost impossible due to an increase of the viscosity, so that 200 ml of deionized water was further added. The resulting aqueous solution had a pH of 9.0.

Then, to the resulting aqueous solution, a zirconium oxynitrate solution was added. After the addition, pH became 5.39.

The zirconium oxynitrate solution was prepared by dissolving 45.67 g of zirconium oxynitrate reagent manufactured by Soekawa Rika in 59.52 g of deionized water.

During the preparation for dropping nitric acid after the addition of the zirconium oxynitrate solution, the stirring was stopped due to an increase of the viscosity. Thus, 1100 ml of deionized water was added thereto.

Then, nitric acid was dropped to adjust the pH of the solution 3.3.

Stirring was continued for a whole day under such conditions. Then, after stopping the stirring, the solution was aged for three days. After completion of the aging, the contents were removed to an evaporating dish to dry at 125°C for 5 hours.

After drying, the dried product was put in a crucible and heated to 535°C at a rate of 7°C/min, followed by calcining for 2 hours.

The calcined Zr/Al catalyst at 535°C was crushed in a mortar and sieved using a 6.5/12 mesh sieve, followed by drying at 125°C for 0.5 hour.

Thus obtained Zr/Al catalyst in an amount of 200.81 g was impregnated in an aqueous solution of Co.

The Co aqueous solution was prepared by dissolving 60.02 g of triethanolamine and 112.61 g of cobalt acetate tetrahydrate in 300 ml of deionized water. The cobalt acetate tetrahydrate in an amount of 112.61 g is equal to 80.06 g of cobalt acetate.

In the course of impregnation in the Co aqueous solution, it was impossible to impregnate the whole solution at a time. Thus, impregnation was conducted for several times. That is, after dipping, the product was dried at 120°C for 10 to 15 minutes; followed by impregnating in the Co aqueous solution. Repeating such a procedure for several times, when the Co aqueous solution was lost, drying was conducted at 120°C for 2 hours, followed by calcining at 450°C to prepare a Co/Zr/Al catalyst.

[DECOMPOSITION OF  $C_2F_6$ ]

Conversion of  $C_2F_6$  was conducted according to the method described in EXAMPLE XVII of USP 6,069,291. The employed gas concentration was 500 ppm as disclosed in EXAMPLE XVII and 5000 ppm conventionally used in the treatment of semiconductor devices.

Experiment 1 (500 ppm)

The above-mentioned Co/Zr/Al catalyst in 6.5/12 mesh in an amount of 41.28 g (considering the size of a reaction tube) was used. The reaction gas composition was 470 ppm of  $C_2F_6$ , 3.2% by volume of  $H_2O$ , and balance being air. The gas hourly space velocity (SV) was  $1800\text{ hr}^{-1}$  and the temperature was  $700^\circ\text{C}$ .

Experiment 2 (5000 ppm)

The above-mentioned Co/Zr/Al catalyst in 6.5/12 mesh in an amount of 39.953 g was used. The reaction gas composition was 5000 ppm of  $C_2F_6$ , 32% by volume of  $H_2O$  (due to use of  $C_2F_6$  concentration 10 times as large as Experiment 1), and balance being air. The gas hourly space velocity (SV) was  $1800\text{ hr}^{-1}$  and the temperature was  $700^\circ\text{C}$ .

[RESULTS]

Attached Fig. 1 shows a relation between the

conversion rate of  $C_2F_6$  at the concentration of 500 ppm and the reaction time.

Attached Fig. 2 shows a relation between the conversion rate of  $C_2F_6$  at the concentration of 5000 ppm and the reaction time.

As shown in Fig. 1, the Co/Zr/Al catalyst of Rossin et al shows high conversion rate of 99% to near 100% for 0 to 2000 hours, when the  $C_2F_6$  concentration was as low as 500 ppm.

On the other hand, when the  $C_2F_6$  concentration was as high as 5000 ppm, the conversion rate of  $C_2F_6$  immediately after the reaction was as relatively low as about 80%, and the conversion rate was lowered with the lapse of time without improving the ability, and lowered to about 60% after 100 hours, and about 35% after 170 hours, as shown in Fig. 2.

The undersigned declarant declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are

punishable by fine or imprisonment, or both, under  
Section 1001 of Title 18 of the United States Code  
and that such willful false statements may jeopardize  
the validity of the application or any patent issuing  
thereon.

Signed this 12th day of September, 2003.

*Shunichi Kanno*

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Shunichi KANNO

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